PATENT ABSTRACTS OF JAPAN

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(54) METHOD FOR PRODUCING GAS SEPARATOR

(57) \bstract:
PROBLEM TO BE SOLVED: To provide a method for producing a simpler gas separator whereby a gas separator which has a higher gas separation efficiency and can give a purified gas with a specified purity can be produced.
SOLUTION: This method for producing a gas separator, by forming a metal film having a gas separation function on a porous substrate, comprises an activation step wherein at least one side of the porous substrate is dipped in a solution containing an activation metal without being accompanied by the pressure difference between the side and the other side and a chemical plating step wherein the one side of the porous substrate is dipped in a solution containing a metal having a gas separation function while being accompanied by the pressure difference between the side and the other side; thus, a metal having a gas separation function is formed into a film on the one side of the porous substrate while blocking minute defects on the surface of the porous substrate.

CLAIMS

[Claim(s)]
[Claim 1]An activation process which is a manufacturing method of a gas separating body with which it comes to form membranes metal which has gas separating ability in a porosity base, and a solution which contains activation metal at least without accompanying a piece side of said porosity base by a pressure differential with other one side is made to immerse.
A piece side of said porosity base is accompanied by a pressure differential with other one side, and it is said gas separating ability.
It is formed metal which is a manufacturing method of a gas separating body provided with the above, and has sa gas separating ability making a minute fault of said porosity base blockade.

[Claim 2]A manufacturing method of the gas separating body according to claim 1 which is an alloy in which metal which has said gas separating ability contains palladium or palladium.

[Translation done.]

* NOTICES *

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to the manufacturing method of the gas separating body which can obtain specific component gas by carrying out diffusive separation of the multicomponent mixed gas. By the simpler production means in details, it is high gas separating efficiency and is related with the manufacturing method of the gas separating body which can obtain high grade specific component gas.
[0002]
[Description of the Prior Art]Conventionally, the membrane-separation method separated by an organic or inorganic gas separation membrane as a method of obtaining only specific gas constituents from multicomponent mixed gas is known. For example as hydrogen separation membrane, the demarcation membrane used for the membrane-separation method has inorganic compound films, such as organic polymer films, such as polyimide and polysulfone, and palladium, or palladium alloy membrane, etc., and has silver or a silver alloy film as a deoxygenation film.
[0003]In particular, in palladium or palladium alloy membrane, it is known that there will also be heat resistance and hydrogen of a high grade can be obtained extremely. The thin film which palladium or a palladium alloy has the character to dissolve and to make hydrogen penetrate, uses this character, and consists of palladium or a palladium alloy is widely used for the gas separating body which separates hydrogen from the mixed gas containing hydrogen. However, there is a problem that a mechanical strength is weak in this palladium membrane independent.
[0004]Then, various proposals have been made in order to solve this problem. In JP,62-273030,A, for example, porous glass, porous ceramics, Or palladium or a palladium alloy is made to laminate on the surface of inorganic porous support, such as a porosity aluminum oxide, and the proposal which raises the mechanical strength of the thin film which consists of palladium or a palladium alloy is made.
[0005]The manufacturing method of the hydrogen segregant which forms palladium membrane in the surface of a heat-resistant porosity base by a chemical-plating method, and heat-treats by forming a silver film by a chemical-plating method, and ranking second on palladium membrane is indicated by JP,3-146122,A. The hydrogen segregant which has a porosity base and a palladium alloy thin film which covers it is obtained, and palladium and silver are uniformly distributed in a palladium alloy thin film by heat treatment.
[0006]The silver film which divides oxygen into U.S. Pat. No. 3,359,705 is indicated.
[0007]However, these gas separating bodies have the problem that the material gas which wears gas separating will leak in purified gas, and will carry out through the minute fault (henceforth a penetration defect) which has penetrated the gas separation membrane which consists of metal which has gas separating ability. Therefore, material gas will mix into purified gas and the purity of hydrogen will fall greatly.
[0008]For example, if the manufacturing method of the hydrogen separation membrane which made the inorganic porous membrane shown in JP,63-171617,A support palladium is explained in full detail, In the gazette, vapor-deposit palladium or a palladium alloy with a SUPPATA ring etc., and an inorganic porous membrane is ranked second, $[Pd(NH_3)_4]$ Carrying out decompression deaeration treatment of the Cl_2 solution via an inorganic porous membrane, evaporating a solvent, and supporting palladium to an inorganic porous membrane is indicated. However, since this hydrogen separation membrane makes nitrogen it not only to to make hydrogen penetrate, but penetrate according to the example, the stoma of the inorganic porous membrane is not blockaded by palladium. Therefore, other constituent gas will be able to mix and the purity fall of hydrogen in purified gas will be caused.
[0009]How to thicken thickness of the gas separation membrane which consists of metal which has gas separating ability as a method of on the other hand preventing generating of such a minute fault can be considered. However, in this method, the problem that the gas permeation nature to which gas penetrates a gas

separation membrane falls, and gas separating efficiency falls may be produced. That is, although the desirable gas separation membrane was a gas separation membrane which there is no penetration defect and can keep the purity of purified gas high and in which gas separating efficiency is excellent, these conditions had a problem of being hard to make it contrary and compatible.

[0010]In order to solve the above-mentioned problem, these people have an activation process and a chemical-plating process, and invented and indicated the manufacturing method of the gas separating body filled up with the metal which has gas separating ability to the stoma currently opened to the surface of the porosity base (patent No. 3213430). The invention is an activation process, and the piece side of a porosity base so that the pressure concerning the piece side may become larger than the pressure of one side of the opposite hand of a porosity base, Make the solution containing activation metal immerse, make by this the inside of a stoma currently opened to the surface in the piece side of this porosity base invade a solution, and at a chemical-plating process. Similarly, the piece side of a porosity base so that the pressure concerning the piece side may become larger than the pressure of one side of the opposite hand of a porosity base, Let the method of filling up a stoma with the metal which has gas separating ability, and making it blockade be one embodiment by making the solution containing the metal which has gas separating ability immerse, and making the metal which has gas separating ability adhere to the stoma of a porosity base.

[0011]According to this manufacturing method, it is possible for the blockade of the stoma of a porosity base to fully be performed, and for material gas to leak in purified gas, not to carry out, and to obtain hydrogen gas of not less than 99.99% of purity. If the depth in which the metal which has gas separating ability has invaded into the inside of the stoma of a porosity base is about 30 micrometers, desired gas separating efficiency can be obtained. Therefore, the gas separating body produced by this manufacturing method is preferably invited to the commercial scene concerning the commercial scene which can solve the above-mentioned problem especially for which the gas of a high grade is needed, for example, a semiconductor manufacturing process, a high-purity-hydrogen purification process, an optical fiber manufacturing process, etc.

[0012]However, the specification demanded depending on a commercial scene is changing in recent years. For example, in the commercial scene concerning the object for mount, or a domestic fuel cell system, in quest of improvement in the purity of the purified gas obtained, specific gravity comes to set to improvement in gas separating efficiency more, and a cost reduction demand is increasing [rather than]. In addition to the aim of miniaturization of equipment concerning gas separating, and it, as the reason, the price jump of palladium in recent years, etc. are cited, for example.

[0013]The gas separating efficiency said to this invention means the gas permeation speed at which gas penetrates a gas separating body, and refers to the amount of gas permeation per time under reference differential pressure. That is, under a fixed supply pressure, since the gas separating body with sufficient gas separating efficiency can be managed with a population smaller although refining separation of the material gas of the specified quantity is carried out, it can reduce facility cost. In a fixed gas separating body population, since the supply pressure required when carrying out refining separation of the material gas of the specified quantity is smaller and it ends, operating costs (electric power cost etc.) can be reduced.

[0014]Therefore, when very high purity was not required of the purity of purified gas, it was expected the manufacturing method which purity is about 99 to 99.9%, for example, and can produce more a gas separating body with higher gas separating efficiency to low cost in the commercial scene which can respond, but. The manufacturing method concerning the above-mentioned patent of these people was not necessarily able to say that it was the optimal to such a demand.

[0015]

[Problem(s) to be Solved by the Invention]There is a place which this invention is made in view of the above-mentioned situation, and is made into the purpose in providing the manufacturing method of a simpler gas separating body which demonstrates higher gas separating efficiency and can produce the gas separating body which can obtain the purified gas which has predetermined purity. As a result of improving each process of the conventional manufacturing method and research piling up in order to meet the demand of a commercial scene, it was found out by the means shown below that the above-mentioned purpose is attained.

[0016]

[Means for Solving the Problem]Namely, according to this invention, it is a manufacturing method of a gas separating body with which it comes to form membranes metal which has gas separating ability in a porosity base, By making a solution containing activation metal immerse at least, without accompanying a piece side of a porosity base by a pressure differential with other one side, By making a solution containing an activation process to which a solution which contains activation metal on the surface in a piece side of a porosity base is contacted, and metal which has gas separating ability for a piece side of a porosity base with a pressure differential with other one side immerse, A manufacturing method of a gas separating body, wherein it is formed

metal which has a chemical-plating process which makes a solution containing metal which has gas separating ability adhere to the surface of a porosity base, and has gas separating ability making a minute fault of the surface of a porosity base blockade is provided. Since work which produces a pressure differential between a piece side of a porosity base and other one side is done only at a chemical-plating process with few routing counters, compared with the conventional manufacturing method, a manufacturing process becomes simpler.
[0017]In this invention, it is preferred that metal which has gas separating ability is an alloy containing palladium or palladium. As for metaled thickness which has the formed gas separating ability, it is preferred that it is about 1-5 micrometers. The gas separating ability of a gas separating body produced by this invention can realize 99 to 99.9% in general as purity of specific component gas produced by dissociating.
[0018]
[Embodiment of the Invention]Although an embodiment is concretely described about the manufacturing method of the gas separating body of this invention below, this invention can add various change, correction, and improvement based on a person's skilled in the art knowledge, unless it is limited to these, and is not interpreted and it deviates from the range of this invention.
[0019]The manufacturing method of the gas separating body of this invention is a manufacturing method of a gas separating body with which it comes to form membranes the metal which has gas separating ability in a porosity base, The film formation condition of the metal which has the gas separating ability in a gas separating body indispensable in order to obtain the purified gas of predetermined purity is defined, and it aims at attaining and having simplification of a manufacturing process and aiming at improvement in a throughput, and reduction of a manufacturing cost, taking care so that it may realize by stabilizing the condition.
[0020]The activation process which the solution which contains activation metal at least in this invention without accompanying the piece side of a porosity base by a pressure differential with other one side so that this purpose may be suited is made to immerse, The chemical-plating process which the solution containing the metal which has gas separating ability for the piece side of a porosity base with a pressure differential with other one side is made to immerse is performed, and it has the feature that it is formed the metal which has gas separating ability making the minute fault of the surface of a porosity base blockade.
[0021]For example, in order to obtain efficiently hydrogen of about 99 to 99.9% of purity as purified gas and to aim at improvement in the hydrogen gas transmission rate of a gas separating body, As for the penetration depth into about 2 micrometers and the porosity base of palladium, it is [the thickness of palladium] preferred that that it is about 1-2 micrometers is metal which has hydrogen gas separability, for example. However, the manufacturing method concerning the patent No. 3213430 of these people was not necessarily able to say that it was the optimal to the demand which such requires, especially controls a penetration depth shallowly with 1-2 micrometers. When the above-mentioned demand is realized, the yield needs to be improved more.
[0022]Although the pressure differential of the piece side of a porosity base and other one side is controlled by the above-mentioned patent of these people at both the processes of activation and chemical plating as the reason, the delicate pressure control for depositing plating shallowly is not easy -- the thickness of palladium, and the penetration depth into the porosity base of palladium -- variation -- I am easy -- things are mentioned.
[0023]Generally an activation process needs to be immersed in two or more medicine, and if the washing process between medicine immersion, etc. are included, it can also turn into ten to 20 process. Therefore, drawing in from one side of a porosity base, if it illustrates more concretely, distinguishing between the piece side and other one side of a porosity base, performing an activation process works more complicated and it causes delay of cycle time.
[0024]In this invention, decompression treatment is not performed by an activation process. In this case, the activation depth, i.e., the depth in which activation metal invades into a porosity base, is automatically set to 1-2 micrometers. Therefore, even if the plating solution which does not control the pressure differential in particular of the piece side of a porosity base and other one side in the following chemical-plating process, for example, contains palladium invades into a porosity base deep, depositing becomes a depth of 1-2 micrometers which is the activated portion.
[0025]In one process of usually impregnating with the metal which has gas separating ability, for example, the plating solution containing palladium, according to this invention, What is necessary is just to control the pressure differential of the piece side of a porosity base, and other one side, and compared with the manufacturing method concerning the above-mentioned patent of these people, work becomes simpler and cycle time can also shorten it. Therefore, a gas separating body can be manufactured more by low cost, and it is easier to accept improvement in gas separating efficiency in a ****** commercial scene than pursuit of purity. It can connect also to evocation of the demand which was not conventionally.
[0026]The predetermined purity of the purified gas in this invention which can be realized is 99 to 99.9% in general. According to this invention, in order to obtain the purified gas of this purity, it is checked that 1-5

micrometers may be sufficient as the metaled thickness which has the formed gas separating ability in general.
[0027]Hereafter, the one embodiment is hung up and each manufacturing process of the manufacturing method of the gas separating body concerning this invention is explained in detail. In the manufacturing method of the gas separating body of this invention, it has an activation process and a chemical-plating process at least. It is not necessary to make the piece side and other one side of a porosity base produce differential pressure in the activation process, as described above. The solution which contains activation metal in the bottom of both atmospheric pressure is made to immerse, and what is necessary is just to contact a solution on the surface in the piece side of a porosity base thereby. By the means of only making it contact in this way, activation metal adheres to the inside of a very shallow stake by the fluid pressure of a solution from the surface of a stoma currently opened on the surface of a porosity base, and the surface of the porosity base. The metal which is the following chemical-plating process and has gas separating ability into the portion to which this activation metal adhered deposits.
[0028]In this activation process, if the piece side is immersed in the solution containing activation metal, one side of that opposite hand does not need to be immersed in a solution. As activation metal, the compound containing palladium divalent ion can be used conveniently. Specifically, the activation process can perform making the hydrochloric acid aqueous solution of a palladium chloride, and a hydrochloric acid aqueous solution with chloridation tin immerse a porosity base by turns.
[0029]Perform electroless deposition using the plating solution containing the metal and the reducing agent which have gas separating ability at least, and the metal which has gas separating ability is made to adhere to the stoma of a porosity base, and thereby, the metal which has gas separating ability fills up a stoma with the following chemical-plating process, and it is made to blockade at it. In this chemical-plating process, one side processed by the activation process is processed. For example, the solution used by the activation process can be transposed to a suitable plating solution.
[0030]In this chemical-plating process, it is preferred to make the metal which has gas separating ability at least, a reducing agent, and the plating solution to contain immerse, as the pressure applied to that one side in the piece side of a porosity base becomes larger than the pressure of one side of the opposite hand of a porosity base. For example, using the porosity base of pipe shape, a plating solution can be made to be able to immerse the outside and the inside of a pipe can be lengthened, for example with a vacuum pump. Using the porosity base of pipe shape, a plating solution may be made to immerse that outside, a pressure may be put on this solution, and the inside of a pipe may be maintained at a fixed pressure. The outside and the inside of this pipe can be made reverse, a plating solution can be made immersed inside a pipe, and, in any case, a pressure can also be changed.
[0031]By making the piece side and other one side of a porosity base produce differential pressure, it becomes easy to make this plating solution invade into the inside of a stoma currently opened to the surface of the porosity base. And electroless deposition of the portion to which activation metal adhered in the previous activation process is carried out. The metal which has gas separating ability is able to adjust the depth which invades from the surface of a porosity base by adjusting the immersion time in a chemical-plating process, the temperature of a plating solution, the pressure differential of both sides concerning a porosity base, etc.
[0032]For hydrogen separation, the publicly known chemical-plating liquid containing silver nitrate, EDTA, an ammonia solution, and hydrazine can be used, for example using the publicly known chemical-plating liquid containing palladium, for example for deoxygenation.
[0033]When producing the gas separating body for hydrogen separation, after carrying out chemical plating of the palladium, it is preferred to heat-treat by carrying out chemical plating of the silver to the electrodeposited palladium surface further, and ranking second to it, to carry out counter diffusion of palladium and the silver, and to alloy palladium and silver.
[0034]The one embodiment is described in detail about the gas separating body hereafter obtained by the manufacturing method of the gas separating body concerning this invention, referring to drawings.
[0035]It is a sectional view showing one embodiment of the gas separating body concerning this invention in drawing 1. The gas separating body 1 is provided with the following.
Porosity base 2.
Metal 3 which has gas separating ability.
Since the porosity base 2 is porosity, it has many stomata 5 in the inside, and there are some which are connected with the surface 6 of the porosity base 2, and are being opened in the stoma 5. The gas separating body 1 obtained by this invention is filled up with the metal 3 which has gas separating ability only for the about six surface [of the porosity base 2 in which the metal 3 which has this gas separating ability touches the gas separation membrane 4 among the stomata 5 currently opened to the surface 6 of the porosity base 2] stoma 5, and blockades.

[0036]As the porosity base 2, as for the material to be used, it is preferred that it is that to which material gas does not react, and, specifically, carbons, porous glass besides a thing, etc., such as alumina, silica, silica alumina, mullite, cordierite, and zirconia, can be used. The thickness in particular of the porosity base 2 is not restricted. What is necessary is just to be able to hold sufficient mechanical strength in an operating environment.
[0037]Although this porosity base 2 has the detailed stoma 5 of a large number which continued in the shape of a three dimension, as for the aperture of this stoma 5, it is preferred that it is 0.003-2 micrometers, and it is more preferred that it is 0.1-1 micrometer or less. It is because resistance in case gas passes in less than 0.003 micrometer in an aperture becomes large. On the other hand, since it becomes difficult to blockade the stoma 5 for the metal 3 which has gas separating ability by chemical plating when an aperture exceeds 2 micrometers, it is not desirable.
[0038]As for the stoma 5 of the porosity base 2, it is preferred that the aperture has gathered. Thereby, do not leak to an about six surface [of the porosity base 2] stoma, it is easy to make a plating solution invade into it uniformly at a chemical-plating process, and the problem which the stoma 5 with which the metal 3 which has gas separating ability is not filled up produces can be avoided.
[0039]The shape of the porosity base 2 of it being a field is preferred, and, naturally also contains with a field the pipe shape equivalent to the shape which included the flat surface and the curved surface, and the curved surface has closed. In the case of pipe shape, the shape of a tube cross section is arbitrary, but a circular thing is easy to receive and it is preferred. Tabular may be sufficient as the shape of the gas separating body 1, or the shape of the porosity base 2, and it is made to arbitrary shape by the purpose of use.
[0040]The metal 3 which has gas separating ability is chosen by gas to refine. For example, they are an alloy which uses palladium and palladium as the main ingredients for hydrogen gas refining, or an alloy containing palladium. In order to separate oxygen, the thin film of the alloy which uses silver or silver as the main ingredients, an organic material thin film, etc. are used.
[0041]It is able to be able to make the gas separation membrane 4 very thin, although the metal 3 which has the gas separating ability which buries an about six surface [of the porosity base 2] stoma by the gas separating body 1 concerning this invention achieves the function of gas separating therefore, and to raise more the gas separating efficiency as the gas separating body 1.
[0042]Although the depth from which the metal 3 which has gas separating ability has invaded into the inside of the porosity base 2 in the gas separating body 1 concerning this invention can be specified if needed, it should just be 1-5 micrometers from the surface 6 of the porosity base 2 in general. It is because a possibility that fear which is not enough will arise, and material gas will be revealed to the purified gas side will increase if this depth is smaller than 1 micrometer. It is because the gas separation membrane 4 exfoliates easily from the surface 6 of the porosity base 2. On the other hand, if this depth is larger than 5 micrometers, gas separating efficiency falls and it is not desirable.
[0043]If the metal 3 which has the gas separating ability which fills up the about six surface [of the porosity base 2] stoma 5 with the gas separating body 1 concerning this invention, and is blockaded is continuously formed with the metal which has the gas separating ability which forms the gas separation membrane 4, in addition, it is preferred. It is because the adhesion of the gas separation membrane 4 and the porosity base 2 improves more and the gas separation membrane 4 becomes difficult to exfoliate from the surface 6 of the porosity base 2.
[0044]When the metal 3 which has gas separating ability consists of palladium alloys, Japanese Membrane Science and 56 (1991) 315-325:"Hydrogen Permeable Palladium- Silver Alloy Membrane Supported. As for the content of metal other than palladium, it is preferred that it is 10 - 30 mass % as indicated to onPorous Ceramics" or JP,63-295402,A. The key objective which alloys palladium is for the improvement in separation efficiency at the time of hydrogen embrittlement prevention and the elevated temperature of palladium. If silver is contained as metal other than palladium, it contributes to hydrogen embrittlement prevention of palladium, and is desirable.
[0045]
[Example]Hereafter, an example is given and this invention is explained still more concretely.
(Example 1) It washed to the porosity base first. It had an outer diameter of 10 mm, 7 mm in inside diameter, and cylindrical shape 300 mm in length, and the fine pore size used for the porosity base the porosity alpha-alumina pipe which is 0.1 micrometer, washed this porosity pipe with water, and, subsequently dried. Subsequently, after holding the solution which contains OPC370 made from Okuno Pharmaceuticals 10% as degreasing treatment at 70 ** and immersing it for 5 minutes, it rinsed.
[0046]Subsequently, activation of the porosity base was carried out. This porosity alpha-alumina pipe was immersed in the solution which contains OPC PURIDIPPINGU 1% for 2 minutes. Next, after holding the solution which contains respectively the inducers A and B made from Okuno Pharmaceuticals 5% at 50 ** and immersing

it for 5 minutes, it rinsed. Next, clyster MU made from Okuno Pharmaceuticals was immersed in the solution included 15% for 5 minutes. Next, clyster MU made from Okuno Pharmaceuticals was immersed in the solution included 3% for 1 minute, and was rinsed. Next, hydrazine was immersed in the solution included 0.05% for 1 minute.

[0047]Since the following another means can also perform activation, only introduction is performed. That is, the 0.1% hydrochloric acid aqueous solution which does 0.1 mass % content of $SnCl_2$ and $2H_2O$ is made to immerse the outside surface of a porosity alpha-alumina pipe for 1 minute. Subsequently, the 0.1% hydrochloric acid aqueous solution which does 0.01 mass % content of $PdCl_2$ is made to immerse the outside surface of this pipe for 1 minute. This dipping treatment is repeated with both hydrochloric acid aqueous solutions so that each hydrochloric acid aqueous solution may be made to immerse 10 times. It is possible to perform activation also by such a means.

[0048]Subsequently, chemical plating of the palladium was carried out. In the removed water 1l, ion $[Pd(NH_3)_4]Cl_2$ and H_2O (5.4g), 2 Na-EDTA (67.2g), the ammonia solution of 28% of ammonia concentration (651.3 ml), preparing the solution which added H_2NNH_2 and H_2O (0.64 ml), and making the above-mentioned solution which carried out temperature control to 50 ** immerse the outside surface of the porosity alpha-alumina pipe which performed activation -- the -- on the other hand, the inside of the pipe was lengthened and decompressed with the vacuum pump. This immersion time was changed and the penetration depth to the thickness of a thin film and the inside of a porosity base which are covered on the surface of a porosity base was adjusted. And yield in[illegible]tigation was conducted about the penetration depth of the palladium which deposited. The accuracy of the penetration depth when a penetration depth is adjusted to 1.5 mm and the thickness of a thin film is adjusted to 2 micrometers was inspected for 20 specimens. The depth inside a porosity base considered as success what is settled in less than 1.5**0.5 mm, and it asked for the yield.

[0049]Subsequently, electroplating of the silver was carried out, and it adjusted so that the weight ratio of palladium and silver might be set to 80:20. Finally it heat-treated by holding at 900 ** for 1 hour, counter diffusion of palladium and the silver was carried out, palladium and silver were alloyed, and the gas separating body was obtained.

[0050]In this way, the pneumatic test was done about the obtained gas separating body. Argon gas was introduced into the alumina pipe exterior, it held by the pressure of 900kPa, and the gas volume revealed to the inside of an alumina pipe was measured.

[0051]The hydrogen separation examination was done about the gas separating body. The mixed gas 17 which consists of hydrogen 80 capacity % and carbon dioxide 20 capacity % was used for material gas. The schematic diagram of test equipment is shown in drawing 2. First, the chamber 7 was heated even at 500 **. Subsequently, the pressure introduced into the outside of the gas separating body 16 the mixed gas 17 which is 900kPa by a part for 2 N l./. The pressure introduced by a part for 0.1 N l./by making argon of 100kPa into the carrier gas 18 in[illegible]e the gas separating body 16. By the gas chromatography, the quantitative analysis was carried out about the purified gas 19 obtained in this way, and the gas permeation speed of the purified gas 19 and the hydrogen concentration in the purified gas 19 were investigated.

[0052]The thermal cycling test was done about the gas separating body. The gas separating body in a hydrogen atmosphere was heated from a room temperature to 500 **, and it ranked second and cooled to the room temperature. This heating and cooling cycle are made into one cycle, and it is 20 cycle *****. The test result of these pneumatic tests, a gas separating examination, and a thermal cycling test is shown in Table 1.

[0053]

[Table 1]

	減圧の有無		ガス分離体			気密試験	ガス分離試験		熱サイクル試験
	活性化工程	化学メッキ工程	膜厚 [μm]	深さ [μm]	歩留まり [%]	漏洩ガス量 [cc/cm²·min]	精製ガス量 [cc/cm²·min]	水素純度 [%]	(50サイクル)
実施例1	無	有	1	1.5	—	0.1以下	138	99.9以上	変化無し
	無	有	2	1.5	95	0.1以下	100	99.9以上	変化無し
	無	有	3	1.5	—	0.1以下	88	99.9以上	変化無し
比較例1	有	無	1	4	—	0.1以下	67	99.9以上	変化無し
	有	無	2	4	—	0.1以下	52	99.9以上	変化無し
	有	無	3	6	—	0.1以下	41	99.9以上	変化無し
比較例2	無	無	1	0.5	—	50	160	70	25回
	無	無	2	0.5	—	20	125	75	31回
	無	無	3	0.5	—	15	80	79	33回
比較例3	有	有	2	1.5	50	0.1以下	100	99.9以上	変化無し

[0054](Comparative example 1) At the process of carrying out activation of the porosity base, in each process

immersed in each solution, the inside of alpha-alumina pipe was made decompression and the gas separating body was obtained according to the palladium chemistry plating process, without using decompression. Since there is no decompression by a palladium chemistry plating process, there is also no implementation of regulation of the penetration depth to the thickness of a thin film and the inside of a porosity base which are covered on the surface of a porosity base, and yield investigation is not conducted. It processed on the same conditions as Example 1 except it. A result is shown in Table 1.

[0055](Comparative example 2) The gas separating body was obtained in the process and both the processes of a palladium chemistry plating process of carrying out activation of the porosity base, without decompressing the inside of alpha-alumina pipe. Since there is no decompression by a palladium chemistry plating process, there is also no implementation of regulation of the penetration depth to the thickness of a thin film and the inside of a porosity base which are covered on the surface of a porosity base, and yield investigation is not conducted. It processed on the same conditions as Example 1 except it. A result is shown in Table 1.

[0056](Comparative example 3) In the process and both the processes of a palladium chemistry plating process of carrying out activation of the porosity base, the inside of alpha-alumina pipe was decompressed and the gas separating body was obtained. With decompression by a palladium chemistry plating process, the thickness of the thin film covered on the surface of a porosity base was adjusted to 2 micrometers. The penetration depth inside a porosity base was adjusted to 1.5 mm, and the accuracy of the penetration depth of the palladium which deposited was inspected for 20 specimens. The depth inside a porosity base considered as success what is settled in less than 1.5**0.5 mm, and it asked for the yield. It processed on the same conditions as Example 1 ex[illegible]ɔt it. A result is shown in Table 1.

[0[illegible]/](Consideration) If the result of Example 1 and the comparative example 2 is compared, in an activation process or a chemical-plating process, by decompressing the inside of alpha-alumina pipe shows that palladium can be adhered even to the inside of a porosity base. When the filling depth from the surface of a palladium alloy increases shows that the airtightness of a gas separating body and the hydrogen purity in purified gas improve. In a thermal cycling test, it turns out that a gas separation membrane becomes more difficult to exfoliate than a porosity base, and adhesion is improving.

[0058]Although it is complicated for it to be immersed in two or more drug solutions at an activation process, and for the routing counter to be able to be 4-20 in general, and to perform decompression treatment in a process respectively, In Example 1, since an effect equivalent to the comparative example 1 is acquired by performing decompression treatment only in one process that a plating solution is impregnated, simplification of a process is possible.

[0059]By comparison of the yield of Example 1 and the comparative example 3, the direction of this invention (example 1) is excellent in delicate thickness control for the deposit depth of palladium into a porosity base to be 1-2 micrometers. In the process and both the processes of a palladium chemistry plating process of carrying out activation of the porosity base, if the inside of alpha-alumina pipe is decompressed, the variation in the deposit depth of palladium will become large, and the yield will fall. If palladium invades too much deeply, for ex[illegible]ple, gas permeation speed will decrease, or if a shallow portion occurs, it will lead to the factor which worsens airtightness. Therefore, undesirably [this yield lowering] in a palladium chemistry plating process, It is useful to make the solution containing the metal which has gas separating ability for the piece side of a porosity base with a pressure differential with other one side immerse, and to control the deposit depth of palladium into a porosity base in the desired depth.

[0060]

[Effect of the Invention]In the manufacturing method of the gas separating body of this invention, only at a chemical-plating process with few medicine used. The piece side of a porosity base so that the pressure concerning the piece side may become larger than the pressure of one side of the opposite hand of a porosity base, A plating solution is made to immerse, and thereby in the surface close part of the stoma currently opened to the surface of the piece side of this porosity base, membranes are formed, the metal which has gas separating ability being filled up with a stoma, and making it blockade. By the activation process using two or more medicine, since it is not necessary to establish the pressure differential of the piece side of a porosity base, and other one side, while work becomes simple, the cycle time of a process is also shortened and a throughput may improve.

[0061]In the gas separating body obtained by the manufacturing method of the gas separating body of this invention, the depth in which the metal which has gas separating ability has invaded into the inside of a porosity base preferably is 1-2 micrometers from the surface of a porosity base, and the thickness of a gas separation membrane is 1-5 micrometers preferably. The gas separating body which fulfills such conditions is excellent in gas separating efficiency, and can contribute to reduction of the facility cost of the gas separating apparatus using this gas separating body, and operating cost.

[Translation done.]

* NOTICES *

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1]It is a sectional view showing one embodiment of the gas separating body obtained by the manufacturing method of the gas separating body of this invention.
[Drawing 2]It is a schematic diagram of the hydrogen separation test equipment concerning the manufacturing method of the gas separating body of this invention.
[Description of Notations]
1 [-- A gas separation membrane, 5 / -- A stoma, 6 / -- The surface, 7 / -- A vacuum chamber, 8 / -- An introducing pipe, 10 / -- An introducing pipe, 15 / -- An O ring, 16 / -- A gas separating body, 17 / -- Mixed ga 18 / -- Carrier gas, 19 / -- Purified gas.] -- A gas separating body, 2 -- A porosity base, 3 -- The metal, 4 which have gas separating ability

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